

NISTIR 6242

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Book of Abstracts
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Kellie Ann Beall, Editor

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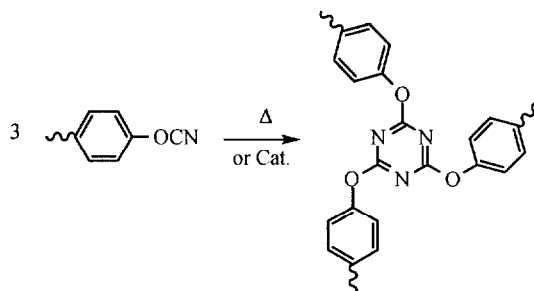
TELECHELIC ARYL CYANATE ESTER SILOXANES AS LOW FLAMMABILITY IMPACT MODIFIERS FOR CYANATE ESTER RESINS

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Introduction

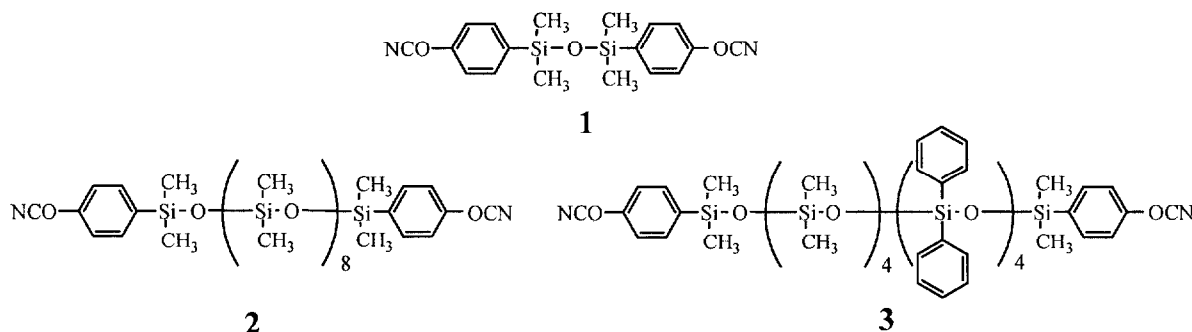
Cyanate ester resins (CERs) have found extensive use in applications where low-dielectric, high thermal stability materials are needed [2]. Polymerization occurs by the cyclotrimerization of aryl cyanate ester groups



These applications include printed wire circuit boards (PCWBs), and radomes. Recently, these resins have been proposed as materials for use as the matrix in structural composites used in civilian air transport. In this application, the organic materials must exhibit low flammability as well as having the appropriate strength and toughness for this type of application. CERs, by themselves possess appropriate flammability properties, but are too brittle for use alone as matrix materials for structure composites. However, their toughness can be greatly enhanced by the introduction of modifiers such as thermoplastics or elastomers. Siloxane based elastomers provide both the requisite improvement in toughness as well as having excellent flame retardancy. To that end, there have been efforts to develop siloxane based impact modifiers for CERs. Previous workers have described approaches utilizing either amine or alcohol terminated siloxanes as impact modifiers[3]. There is also a patent issued for the development of allyl(2-phenylcyanate) terminated siloxanes[4]. In the former case, there is concern that the cross-linking reaction may introduce polar functionality into these materials causing excessive moisture absorbance. In the latter materials, the added hydrocarbon component of the allyl group is a potential problem in terms of combustability of the siloxane. We have focussed our attention on developing a family of telechelic siloxanes terminated with p-cyanatophenyl groups.

Results and Discussion

We were able to synthesize the a variety of cyanate-ester functional siloxanes in high yield[5].



Homopolymerization of compound 1 by curing from 120-280°C leads to a glassy solid with T_g of 274°C. For compound 2, the result is a rubber with a T_g of -110°C and for compound 3 a rubber with a T_g of 8°C. All three compounds exhibit a thermal decomposition at roughly 500°C as observed via TGA. The longer chain siloxanes also exhibit a loss peak at 200°C. Commercial CERs show a loss only at 500°C. We interpret this to mean that the 500°C loss is due to breakdown of the triazine and/or ether linkages.

When compound 1 was blended with the commercial CER Primaset PT-30 (a novolac-based CER from Lonza) at 10 phr, the resultant cured material exhibited a single phase. However, for blends with compounds 2 or 3, a two-phase material

is formed. In both case the oligomer was not soluble and agitation during the initial stages of cure was required. For the blend with **2**, the morphology of the blend indicates that the siloxane forms the continuous phase, while for the blend with **3**, the PT-30 is the continuous phase. For the blend with the diphenylsiloxane containing additive (**3**), the typical domain size of the rubbery phase is 20-30 μm (**Figure 1**).

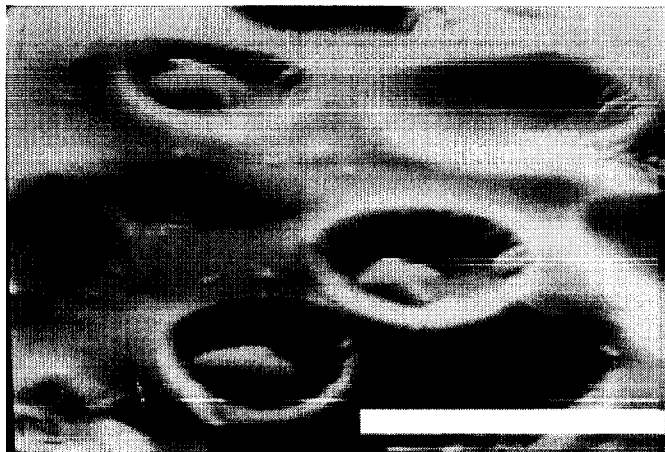


Figure 1. SEM photograph (100,000 x) of fracture surface of 10 phr telechelic siloxane **3** in PT-30 after curing. (inset bar is 50 μm)

Conclusions

We have demonstrated the synthesis of a number of telechelic CERs which can be compounded with commercial CERs to form either single or two phase blends. In the case of the two-phase blends, the rubbery phase domain size is in the range necessary for improving the fracture toughness of the commercial resin. We are currently exploring the effect of oligomer composition and molecular weight on the mechanical and flammability properties of these new materials. We are also examining the thermal and mechanical properties of cured blends of the disiloxane CERs with oligosiloxane CERs.

Acknowledgements

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